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Surface layer modification of 12Cr1MoV and 30CrMnSiNi2 steels by Zr+ ion beam to improve the fatigue durability

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Abstract

With the use of optical/scanning electron microscopy and microindentation some peculiarities of structure modification over cross-section as well as mechanical properties of 12Cr1MoV and 30CrMnSiNi2 steel specimens subjected to ion-arc treatment by Zr ion beam were investigated. It is shown that changing of the microstructure takes place over entire cross-section that is not the case of material softening due to annealing. Within the revealed results on structure modification the data on changing mechanical properties under cyclic and static tension of the irradiated specimens are interpreted. Of particular interest are results on strain measurement by digital image correlation technique.

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Keywords: microindentation; cyclic and static tension; structure modification; strain measurement.

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1. Introduction

The overwhelming majority of structure elements and machine parts under operation experience influence of variable loadings that might be the reason of their fatigue fracture. The problem of studying and predicting fatigue fracture keeps urgency and contains a number of unsolved scientific and engineering problems [1].

Surface nanostructuring is the effective way of increasing fatigue strength and wear resistance of machine parts. Treatment of surface layers is of special actuality for fatigue fracture where the state and defects in surface layer plays the key role [2] In doing so design of advanced techniques for surface modification is always a strict compromise between enhancement of surface strength and providing high enogh ductility in order to diminish the negative role of stress concentrators to occur there due to presence of scratches or microcracking to take place under cyclic mechanical loading.

The aim of the study is to investigate the effect of surface irradiation of heat-resistant 12Cr1MoV and 30CrMnSiNi2 high strength steels by Zr +ions onto increase of their fatigue durability. 12Cr1MoV steel belongs to a class of heat resistant ones, and is intended for manufacturing the machine parts to operate at high temperatures of 540-580°C in particular, for steam-pipe lines. Another steel under investigation was 30CrMnSiNi2. It is not heat resistant one, so as a result of the ion-beam treatment its mechanical properties can significantly degrade. In addition, introduction of Zr+ ions into a thin surface layer as one more alloying element, should lead to complication of its structural-phase composition, and probably to the embrittlement. In doing so, a special regime that includes a rotation of specimens during the treatment was developed that allows to remove specimens from the impact of the ion-beam thereby reducing their thermal softening.

2. Material and methods

12Cr1MoV steel was used as model material since it does not experience structural changes at the temperature at which the process of surface layer nanostructuring by ion beam is carried out. Besides, the steel is quite ductile, so the study of processes of localized deformation and fracture under cyclic load provides much more evidence at lower rate of deformation processes. High strength 30CrMnSiNi2 steel is used for manufacturing responsible and highly loaded parts that experience action of alternating loadings. Increasing of fatigue durability of this steel is a complex problem because of it‘s low heat resistance and high level of alloying.

Flat specimens with of size 70×10×1 mm were prepared from a 12Cr1MoV steel pipe fragment by electro-spark cutting. For running fatigue tests the holes of 2 mm were mechanically drilled as a stress concentrator at a distance of 50 mm from one of its edges. For static tension tests of 12Cr1MoV steel dog-bone shaped specimens with gauge length of 20×5×1 mm were also employed, as well as ones with the stress concentrator for 30CrMnSiNi2 steel (similar to the specimens for fatigue tests). The specimens were mechanically polished and divided into 2 groups: a) in the as-supplied state (without the treatment) and b) ones with a surface layer irradiation by zirconium Zr+ ion beam.

Ion-arc treatment (nanostructuring) of surface layer was carried out with a help of high current vacuum-arc source of metal ions UVN-0.2 "Quant". The microstructure of specimens after chemical etching by 5 % solution of nitric acid was investigated. Micrographs of specimen’s surface were obtained by means of optical microscopes Carl Zeiss Axiovert 25 CA and Carl Zeiss EPIQUANT, as well as scanning electron microscope Carl Zeiss EVO 50. Surface profilometry were performed with the help of optical interferometer of white light NewView 6200. X-ray phase analysis was conducted by X-ray difractometer DRON-7.

Static tension tests were performed with the use of electromechanical testing machine Instron 5582 while for the cyclic tension tests of 30CrMnSiNi2 steel servohydraulic testing machine Biss UTM 150 was employed. Specimens of 12Cr1MoV were tested under cyclic alternating cantilevered bending with frequency of 10 Hz. Surface micrographs of specimens were captured by Canon D550 digital photo camera during the process of cyclic loading to estimate strain distribution by optical technique (DIC) as well as to characterize strain induced relief. Microhardness was estimated by PMT-3 microhardness meter with the load of 0.5 N.

Local strain was estimated through calculating of normalized value of shear strain intensity (principle strain) with the use of Digital Image Correlation technique. In doing so, optical micrographs were captured during the cyclic loading with further construction of displacement vector fields (DVF) and their numerical differentiation.

3. Experimental results

3.1. Study of the modified surface layer of 12Cr1MoV steel

The structure of the surface layer of the 12Cr1MoV steel in the as-supplied state is represented by large ferrite grains with average diameter of >1 µm with inclusions of cementite (Fe3C). Surface layer structure after nanostructuring by the Zr+ ion beam is represented by phases of FeZr3, FeZr2, as well as ferrite grains. The average size of the grains in the surface layer makes 100-150 nm.

The microhardness over the irradiated specimens crossection was measure. On the basis of obtained results one can judge on the thickness of the subsurface layer to be softened during Zr+ ion beam irradiation. It was estimated to be equal to 100-150 µm. Simultaneously, at the depth more than 100 µm the microhardness was increased by 22 % in contrast with specimen in the as-supplied state.

With the help of 5% solution of nitric acid the microstructure of the specimens in both states were examined. It is seen from Fig. 1 that it consists of ferrite-perlite grains. In order to estimate microstructure changes through the thickness the lateral face of the specimens were polished (Fig. 1,b). One can distinguish the difference in structure within subsurface layer and bulk material. The thickness of the modified layer can be estimated as being equal to 90-130 µm while it does not have pronounced boundary with adjacent bulk layers. As is known from pyrometer measurement data this layer experiences short term heating up to the temperature of 850° C that can give rise to grain growth. At the same time the core of the specimen is subjected to such heating in a less degree. The rotation of the specimens in the vacuum chamber during the irradiation (cyclic heating with further cooling when specimen does not contact with the ion beam) combined with possible carbon diffusion from the subsurface layer might give rise to certain decrease of grain size from 30-50 µm down to 20 µm that somehow becomes visible from Fig. 1,b. This might be a possible explanation of microhardness increase in contrast with non-treated specimens.

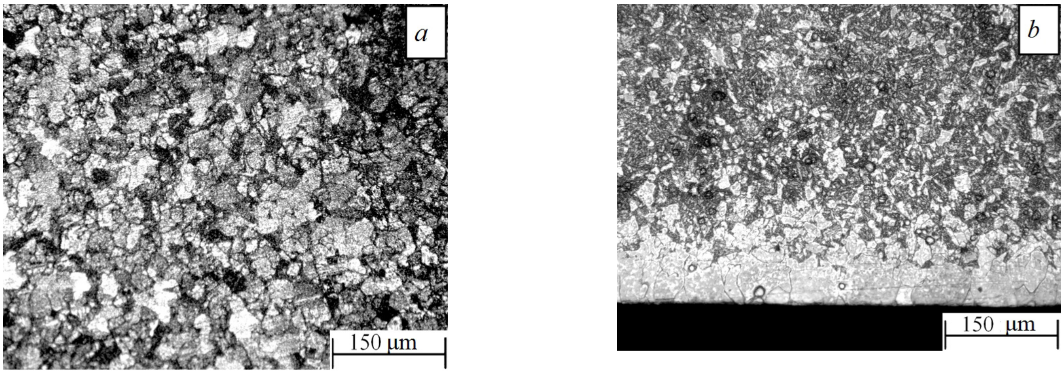

Fig. 1. Optical micrographs of etched surfaces of specimens in the as-supplied state (a) and after the nanostructuring by Zr+ ion beam (b)

3.2. Study of the modified surface layer of 30CrMnSiNi2 steel

In contrast with above discussed heat resistant steel the high strength 30CrMnSiNi2 steel experience quite different structure changes. In the subsurface layer at the distance not exceeding 6 µm from the surface intermetallic compounds of Fe-Zr system are formed as well as zirconium carbides. Then, at the thickness up to 35 µm due to action of elevated temperatures to occur under irradiation the tempering takes place that gives rise to ferrite-cementite structure formation with characteristic grain size of 2-5 µm. In the deeper layers located at the distance more than 100 µm from the surface the sorbite structure is formed that is characteristic feature for for high temperature tempering to take place at heating above 650 °C. Because of thermal cycling to occur during the ion-beam irradiation the residual austenite transferes into the beinite that is responsible for beinite-martinstite structure to be found at the distance more than 100 μm. The latter ensures certain increase of the microhardness in contrast with austenite-martensite structure being characteristic for thermal treatment at standard parameters.

3.3. Static tension tests of 12Cr1MoV steel

During the tensile tests the loading diagram of 12Cr1MoV steel of dog-bone shape specimens was registered. It was found that for nontreated specimens the presence of sharp yield point (yield tooth) is evident much like it takes place for low carbon steels. Yield point of such specimens makes σ_{ys} = 270 ± 25 MPa, ultimate strength – σ_{us}= 494 ± 36 MPa and elongation – ε = 20 ± 3 % which is close by values to the reference book data for this steel [3]. After the surface nanostructuring the value of ultimate strength is increased up to σ_{us} = 570 ± 17 MPa while elongation becomes lower ε = 16 ± 0.7 %. In doing so, there is no formation of the yield plateau at the diagram of the processed specimens.

3.4. Static tension tests of 30CrMnSiNi2 steel

The tests on the static tension of the specimens with the central holes were carried out to have the same shape as ones used under cyclic tension. It was shown that the specimens without the treatment had the ultimate strength of σ_{us} = 1628 MPa, relative elongation ε = 6 %; the specimens subjected to the ion modification had the ultimate strength σ_{us} = 1270 MPa, relative elongation ε = 8 %. Thus, as a result of the ion treatment of the 30CrMnSiNi2 steel the ultimate strength was lowered by 22 %, relative elongation increased by 25 %.

3.5. Test on cyclic alternating bending 12Cr1MoV

According to the testing data the fatigue life-time of specimens under cyclic alternating bending is increased due to the structure modification by~ 2 times. The series of optical micrographs were used to calculate and plot the graphs to characterize dependence the crack length versus the number of loading cycles.

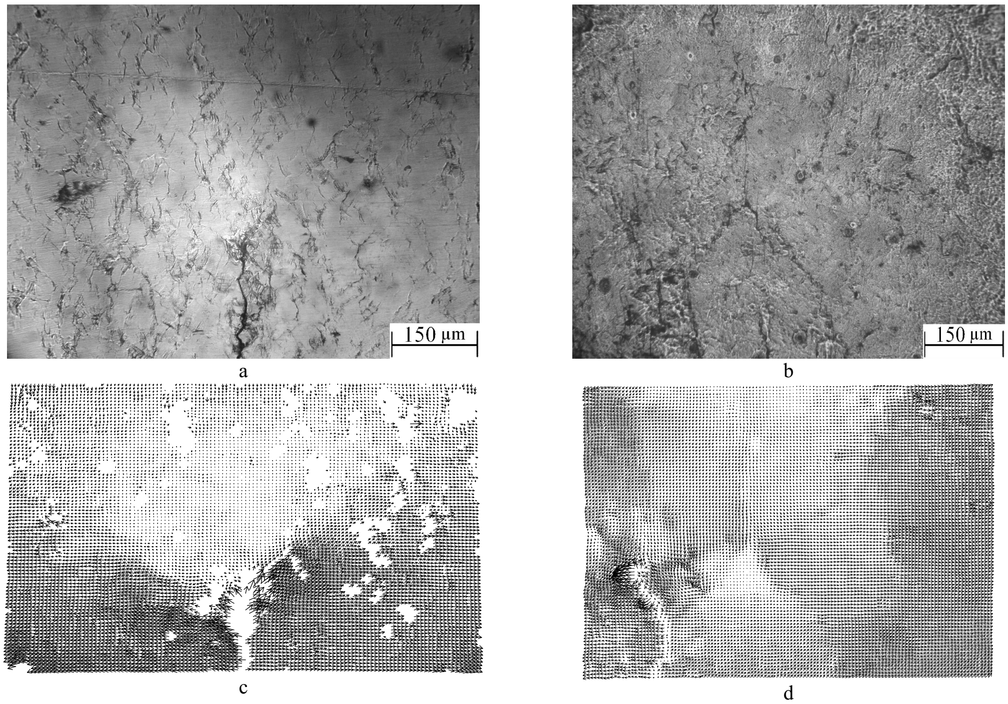

Fig. 2. Optical micrographs (a,b) and corresponding displacement vector fields (c,d) in specimens under cyclic bending; a),c) specimens without the treatment; b),d) after the nanostructuring by ion beam irradiation; a),c) 3000-4000 cycles; b),d) 3000-4000 cycles

It is shown that under cyclic bending the main crack originates nearly at the same time for both types of specimens but has significant differences in the propagation rate. Crack growth rate for the untreated specimen was 0.05 μm/cycle while for the specimen with modified surface layer it made 0.024 μm/cycle. Thus, it is shown that nanostructuring of surface layer by ion beam to slow the rate of fatigue crack growth by about 2 times. The measurement of surface roughness of failure specimens in the fracture area was carried out with a help of the optical interferential profilometer. It is shown that values of the surface roughness parameter Ra of the specimen the without the treatment is more than 1.5 times higher in comparison with one for the specimen after the irradiation. During the test, in the specimen without the treatment clearly pronounced microcracks are formed along the grain boundaries and the main crack is more clearly manifested to propagate just along the microcracks. The specimen after the treatment has the number and size of the microcracks noticeably smaller and the main crack develops to a less extent. The main crack in the irradiated specimen is less pronounced that could indicate that it develops mainly in the modified surface layer which hinders its spreading into the bulk of the material. The analysis and comparison of optical micrographs and displacement vector fields gained during cyclic bending tests allowed revealing differences in patterns of deformation distribution at fatigue crack opening (Fig. 2). Under testing of the nontreated specimens regardless the crack branching it develops by its faces opening by normal mode that is accompanied by formation of a couple of meso shear bands to originate at the crack tip (Fig. 2,a,c). At cyclic bending of the treated specimen it is difficult to reveal exact crack paths (Fig. 2,b) since presence of the modified layer disperse stress concentrator in the crack tip and deformation in its vicinity is redistributed (dispersed) over larger area. This becomes evident at observation of displacement vector field (Fig. 2,d): it is clear that some regions with self-consistent displacements are formed that tend to accommodate rotational deformation modes to occur at crack propagation. This might be resulted from difference in strength of modified surface layer and substrate since they possess different ductility as well. Being based on data of microhardness measurement one can postulate that softening of the subsurface layer can play a positive role at increasing the fatigue life time since crack arrest effects can occur in more ductile modified subsurface layer of several tens μm thickness.

3.6. Cyclic tension tests of 30CrMnSiNi2

During the tests the average number of cycles prior the fracture were determined. For the specimens without the treatment this value made Np =110 000±31 000 cycles while for the specimens after the irradiation – 330000 ± 40 000. The specimen after tempering failed after 138 000 ± 36 000 cycles. Thus, the surface modification by the Zr+ ion beam irradiation of 30CrMnSiNi2 steel specimens ensures increasing the fatigue life-time by 3 times.

The dependence graph of the fatigue crack length vs the number of loading cycles was built being based on the surface image analysis captured during the tests. It is evident that the crack starts substantially later and develops slower in the specimen after the ion-arc treatment. For the specimens without the treatment the rate of the crack growth made L = 0.103 μm/cycle while after the ion treatment L = 0.025 μm/cycle. Thus, the surface layer modification of the specimens by the Zr+ ion beam contributes to the delay of the fatigue crack nucleation by ~3 times, as well as increases the time of its propagation by ~4 times. The softened subsurface layer provides more uniform distribution of stresses and “heals” dangerous defects on the surface.

Displacement vector fields were constructed with the use of optical images registered during cyclic tension tests. In Fig. 3 optical micrographs for non- and irradiated specimens gained at the pre-fracture stage are given. It is seen that in the specimen without the treatment plastic deformation is localized in the tip of fatigue cracks being propagated from the stress concentrators (Fig. 3,a). The growth of cracks is accompanied by plastic deformation which is also manifested by shrinkage of the specimen (Fig. 3,b).

In general it can be concluded that the deformation behavior of the specimen with a couple of fatigue cracks is determined by their gradual propagation and visco-elastic response of the surrounding material to reduce the cross-sectional area of the specimen in this region. A quite different pattern is observed in the specimen after the treatment where near-surface layer is softened at a thickness of 100 μm. Much like the specimen without the treatment a fatigue crack grows from the stress concentrator (Fig. 3,b); but its propagation is accompanied by intensive deformations in the area in the vicinity of the hole, that can be the reason of dispersing the powerful stress concentrator due to the involvement of a large number of mesoconcentrators of lower power.

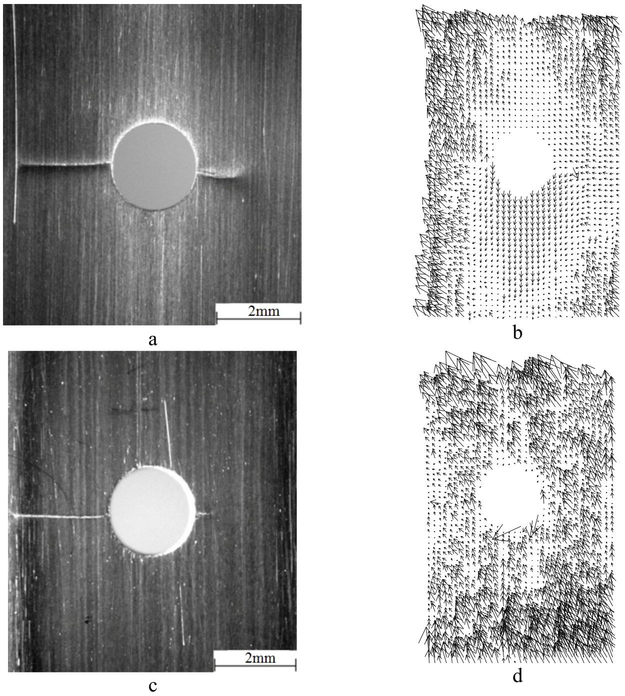

Fig. 3. Surface optical micrographs (a,c), and displacement vector fields of specimens (b, d): a), b) – in the as-supplied state (100×103 cycles); c), d) – after treatment with Zr+ ion beam (384×103 cycles)

As a result, instead of propagation of the cracks by the normal fracture mechanism for the case of specimen without treatment, there is quasi-homogeneous distribution of mesoscopic deformation take place (Fig. 3,d). This may be the reason for the fatigue crack growth damping and fatigue durability increase [4,5].

4. Summary

The structure investigation of subsurface layer and cross section of 12Cr1MoV and 30CrMnSiNi2 steel specimens after the ion-beam irradiation has been carried out. It is shown that that the treatment brings to the formation of the softened subsurface layer with approximate thickness of 100 μm. The core of the specimen also experience the changing of the hardness that is increased by 22 % for 12Cr1MoV steel while for 8 % for 30CrMnSiNi2 one. The tests have shown that the ion-beam treatment used ensures the increase of fatigue durability by 2-3 times in contrast with the non-treated specimens.

At cyclic alternating bending of 12Cr1MoV steel the main crack origination in specimens of both types happens at nearly equal numbers of cycles that is associated with the periodic occurrence of tensile and compressive stresses in the surface layer. However, at the subsequent stage of steady fatigue crack growth the main crack propagation rate is about 2 times lower since the softened surface layer ensures effective redistribution of stresses, as well as dispersing stress concentration at the grain boundaries. This lowers stress intensity in the vicinity of main fatigue crack tip.

Analysis of strain fields of 30CrMnSiNi2 steel allows revealing the influence of modified surface layer to provide efficient redistribution of deformation resulting in much later nucleation of the main crack and its slower propagation. This leads to the increase of fatigue durability of specimens after the treatment. The main reason for the revealed changes is softening of the subsurface layer since in contrast the quenched steel specimen are very sensitive to presence and nucleation of microcracks whose evolution usually is completed with fast nucleation and propagation of main fatigue crack.

Acknowledgements

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